

Technical Report Documentation Page

1. REPORT No.

2. GOVERNMENT ACCESSION No.

3. RECIPIENT'S CATALOG No.

4. TITLE AND SUBTITLE

Analysis of Cooperative Data for Optimum So₃ by 24-Hour
Compressive Strength Tests of One Cement

5. REPORT DATE

December 1959

6. PERFORMING ORGANIZATION

7. AUTHOR(S)

W.E. Haskell

8. PERFORMING ORGANIZATION REPORT No.

9. PERFORMING ORGANIZATION NAME AND ADDRESS

State of California
Department of Public Works
Division of Highways

10. WORK UNIT No.

11. CONTRACT OR GRANT No.

12. SPONSORING AGENCY NAME AND ADDRESS

13. TYPE OF REPORT & PERIOD COVERED

14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

This report discusses results obtained with one cement, as a part of a cooperative program of testing being conducted by the Working Committee on SO₃ Content of ASTM C-1. The objective of the program is to investigate the compressive strength of Ottawa sand mortars at the age of 24 hours as a means of (1) determining the optimum SO₃ content of the cement, and (2) by tests of mortars containing the cement with a single addition of gypsum, measuring the relationship of the SO₃ content of the cement as received to its optimum value.

Instructions for making the tests as given by Mr. T.B. Kennedy, Chairman of the Working Committee, are given in the appendix.

The portions of the work covered by this report were made on a sample of Type II, low-alkali cement furnished by Calaveras Cement Company, San Andreas, California. The laboratories cooperating in the test program with this cement were: Ideal Cement Company

17. KEYWORDS

18. No. OF PAGES:

32

19. DRI WEBSITE LINK

<http://www.dot.ca.gov/hq/research/researchreports/1959-1960/59-25.pdf>

20. FILE NAME

59-25.pdf

M & R
EH/12/59

3988
C. 2

LIBRARY COPY

Materials & Research Dept.

STATE OF CALIFORNIA
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

ANALYSIS OF CO-OPERATIVE DATA

FOR

OPTIMUM SO_3 BY 24-HOUR COMPRESSIVE STRENGTH

TESTS OF ONE CEMENT

LIBRARY COPY

Materials & Research Dept.

By
W. E. Haskell
Associate Materials and Research Engineer
California Division of Highways

December 29, 1959

59-25



Analysis of Co-operative Data for Optimum SO_3
by 24-hour Compressive Strength Tests of One Cement

By
W. E. Haskell
Associate Materials and Research Engineer
California Division of Highways

This report discusses results obtained with one cement, as a part of a co-operative program of testing being conducted by the Working Committee on SO_3 Content of ASTM C-1. The objective of the program is to investigate the compressive strength of Ottawa sand mortars at the age of 24 hours as a means of (1) determining the optimum SO_3 content of the cement, and (2) by tests of mortars containing the cement with a single addition of gypsum, measuring the relationship of the SO_3 content of the cement as received to its optimum value.

Instructions for making the tests as given by Mr. T. B. Kennedy, Chairman of the Working Committee, are given in the appendix.

The portions of the work covered by this report were made on a sample of Type II, low-alkali cement furnished by Calaveras Cement Company, San Andreas, California. The laboratories co-operating in the test program with this cement were:

Ideal Cement Company
Research Laboratory
Fort Collins, Colorado
Kenneth E. Palmer

Marquette Cement Manufacturing Co.
Research Laboratory
Chicago, Illinois
O. E. Brown

California Division of Highways
Materials and Research Department
Sacramento, California
Bailey Tremper

The program in brief, as it was conducted, consisted of the molding and testing of 2-inch Ottawa sand mortar cubes at 24 hours. Additions of pulverized gypsum (terra alba) were made to the cement to result in SO_3 contents of 1.00, 1.50, 2.00, 2.50, and 3.00 per cent. Each of the prepared cements were mixed in 1:0.5, 1:1 and 1:2 mortars having a flow between 100 and 115. Two rounds were made on separate days. At the conclusion of these tests each co-operator estimated the optimum SO_3 content of the cement and made three additional rounds with cements calculated to contain -0.25, 0, and +0.25 per cent SO_3 relative to optimum. California Division of Highways also made one round of 1:2 mortars upon which expansion in water and contraction in air were determined.

Data of the Tests

Data of the tests are given in the following tables and figures.

Table I. Chemical analysis of the cement as performed in two laboratories. For the purpose of this report the SO_3 content is considered to be 1.00 per cent.

Table II (a), (b) and (c). Original test results for compressive strength and derived data showing differences by increments of SO_3 .

Table III. Original test results and derived data for expansion and contraction.

Table IV. Derivation of parabolic equations representing SO_3 -strength relationship, the coefficient of correlation and the standard error of estimate for the results of each laboratory.

Table V (a), (b) and (c). Mathematical computations based on strength data, of optimum SO_3 as indicated by each batch of mortar at each level of SO_3 and departures from "true" optimum.

Table VI. Mathematical computations based on contraction data, of optimum SO_3 for single batches of mortar at each level of SO_3 and departures from "true" optimum.

Table VII. Data of the precision of compressive strength tests in relationship to change in strength produced by an increment of 0.5 per cent SO_3 . Also data of the precision of the contraction test abstracted from a previous report of cooperative tests.²

Figure 1. Parabolic curves showing SO_3 -strength relationship for 1:0.5 mortar.

Figure 2. Similar curves for 1:1 mortar.

Figure 3. Similar curves for 1:2 mortar.

Figure 4. Parabolic curves showing SO_3 -contraction relationships for 1:2 mortar.

Discussion

The determination of optimum SO_3 from freehand curves drawn to fit points representing the SO_3 -strength relationships may not be the best fit. Furthermore, unless such curves can be defined by a mathematical equation, it is difficult to determine values at points between those representing experimental results.

It has been found¹ that data of SO_3 versus contraction can be expressed quite well by a parabolic equation. The equation of a parabola is easy to handle mathematically and is used in this report to express the SO_3 -strength relationships. Parabolic equations have been computed for the three mortar mixtures used in this study. The curve representing change in strength produced by an increase in SO_3 of 0.5 per cent, is parallel to the locus of tangents to the parabola; i.e., to the first derivative of the parabolic equation, and is a straight line. The determination of the best fit of a straight line to experimental data by the method of least squares is relatively simple. Such computations have been made for the strength data of this study using individual strength results for each batch of mortar. The results are shown in Table IV. Having

determined the equation of the straight line, the equation of the parabola can be computed by integration. This is quite simple if the co-ordinates are adjusted so that zero on the abscissa represents optimum SO_3 and the ordinates are shown in terms of relative strength.

The equation representing the change in strength is of the form

$$n = m (g + 0.25) + k \quad (1)$$

and the equation of the parabola is:

$$s = m g^2 \quad (2)$$

where n = change in compressive strength, pounds per square inch per unit (1 per cent) of variation in SO_3

m = a coefficient dependent on the characteristics of the cement and on test conditions (different values of m are obtained for each mix proportion).

g = percentage of SO_3 in the cement relative to its optimum value.

k = a constant dependent on test materials and conditions. It is not necessary to determine the value of k in order to use the equations in the manner to be described.

s = relative compressive strength in pounds per square inch.

Having determined by least squares the value of m in equation (1), equation (2) is readily written by using the same value of m .

For example, the value of the coefficient m for the 1:0.5 mix has been computed to be -1170. The value of g for

any measured change in strength can then be computed by rearrangement of equation (1). If the measured change in contraction for the cement containing 1.00 per cent SO_3 is +1144, the applicable equation is

$$g = \frac{1144}{-1170} - 0.25 \quad (3)$$

and $g = -1.23$

This result indicates that the cement is deficient in SO_3 by 1.23 per cent and the optimum is 2.23 per cent.

Figures 1, 2 and 3 are graphical representations of the curves with reference to the experimental data given in Table IV. Plotted points are shown for average values of strength only. It will be noted that the best fit of the curves is obtained with the 1:2 and 1:1 mixes and that the fit for the 1:0.5 mix is considerably poorer.

Table IV shows the computed values of the coefficient m , and the coefficient of correlation, r , for the individual laboratories and the group data. The correlation coefficients for the group data are all significant at the 0.1 per cent significance level (99.9 per cent confidence level), but the coefficients for the 1:1 and the 1:2 mixes are larger than for the 1:0.5 mix, thus indicating a greater significance. The standard error of estimate is lowest for the 1:2 mix, is slightly higher for the 1:1 mix and is decidedly higher for the 1:0.5 mix. This function is a mathematical expression of

the variation of the data about the line of best fit, as shown graphically in Figures 1, 2 and 3.

Computations of the precision of the test data in terms of repeatability and reproducibility are given in Table VII. The cement with each increment of SO₃ is treated as a separate cement and the precision is computed for the values of change in strength for an increment of 0.5 per cent SO₃ at each level of SO₃ content. The average precision for the four levels of SO₃ content is shown in the next to last column.

The last column gives values of the "precision ratio". It is a measure of the extent to which the repeatability or reproducibility is larger or smaller than the change produced by the addition of 0.5 per cent SO₃ or:

$$\text{Precision Ratio} = \frac{\text{Repeatability or reproducibility}}{\text{Rate of change produced by 0.5\% SO}_3}$$

It is computed by dividing the average repeatability or reproducibility by one-half the value of m . Since repeatability and reproducibility refer to the greatest differences between two independent results (within laboratories or operators, and between laboratories) that are not significantly different at the 95 per cent probability level, the precision ratio simply states whether the addition of 0.5 per cent SO₃ will produce a change greater or less than the largest difference between two results that are acceptable on the basis of a given statistical probability; in this instance, the probability of 95 per cent. The data indicates that the 1:0.5 mix gives

distinctly poorer results than the other mixes. The data available for computing measures of precision (single pairs of tests from three laboratories) are the minimum that can be justifiably used. A larger volume of data would give greater reliability.

Table VII also contains values of repeatability and reproducibility (hereinafter referred to as precision indexes), for change in contraction as given in a report of the Working Committee on SO_3 Content². In this study, it was found that the precision was much better when the test cement contained SO_3 near its optimum value which is the condition to be expected in routine shipments. Therefore the precision indices for the cement content nearest optimum are given. The precision ratios for the contraction test are somewhat lower and therefore more favorable than those for the strength test. However, the comparison as made is unfavorable to the contraction test for the reason that the value assigned to the coefficient of contraction, p , is the average of that found for four cements, whereas the coefficient, m , applied to the strength tests, is that which applies to a single cement only. Furthermore, for the cement used in the present study, the coefficient of contraction is 0.021. If the precision ratios are calculated with one-half of this value as the denominator, they become 0.52 for repeatability and 0.44 for reproducibility.

The precision of the tests can be estimated in another way and the results are shown in the last column of Table V.

This gives the difference in the estimate of optimum SO_3 derived from a single pair of tests and the "true" value of optimum as determined by all tests for the particular mix being considered. The percentage of cases where the difference exceeds 0.25 is given below.

1:0.5	42 per cent
1:1	0 per cent
1:2	25 per cent

These results indicate that the 1:1 mix gives the greatest accuracy. Furthermore it should be noted that the majority of instances where differences in estimate exceeded 0.25 per cent, the SO_3 content was in the region of optimum.

The results of the computations for the significance of the correlations and the precision of the test data, may be summarized as follows.

(1) The coefficient of correlation for the equations representing the SO_3 -strength relationships are the poorest for the 1:0.5 mix.

(2) The standard error of estimate for the equations representing the SO_3 -strength relationships is poorest for the 1:0.5 mix.

(3) The precision ratio which expresses values of repeatability and reproducibility in terms of the coefficient m of the change in strength equation is poorest for the 1:0.5 mix.

(4) Estimates of optimum SO_3 from single pairs of tests are poorest for the 1:0.5 mix. On the whole, the 1:1 mix appears to yield values of greater accuracy than does the 1:2 mix.

Based on values of repeatability and reproducibility, none of the strength tests yield the precision obtainable with the contraction test if cements containing SO_3 contents in the region of optimum are considered.

It should be emphasized that all conclusions reached in this study are based on results of tests of one cement only. They may require substantial modification if results of tests of other cements are similarly analyzed.

The apparent value of optimum SO_3 as determined by the several tests are not constant. Values are listed below.

Strength Test	1:0.5 mix	2.30 per cent
"	" 1:1 mix	2.27 per cent
"	" 1:2 mix	2.24 per cent
Contraction Test	1:2 mix	2.07 per cent

Differences in optimum SO_3 as determined by the several strength tests are not significant; however, the value determined by the contraction test is lower by 0.20 percentage point. It should be borne in mind however, that optimum by the contraction test was determined from data of a single round in one laboratory. The result may be in error and the true difference may not in fact be as great as indicated.

In addition to the strength tests discussed above, each

laboratory made three additional rounds with the cement calculated to contain -0.25, 0 and +0.25 per cent SO_3 relative to optimum. Values of optimum evidently were selected from the freehand curves of the SO_3 -strength relations and the values as selected by the different laboratories did not agree exactly. Theoretically the strengths at -0.25 percent SO_3 and at +0.25 per cent SO_3 should be identical, while that at optimum should be slightly greater. The precision of the tests is not high enough to establish these relationships with certainty. The data were not helpful in the overall analysis.

References

1. Bailey Tremper, "The Control of Gypsum in Portland Cement", Proceedings ASTM, Vol. 59 (1959)
2. "Short-Time Tests of Mortars Controlling SO₃ in Portland Cement at Optimum Value", reported by Working Committee on SO₃ Content of ASTM Committee C-1. Available from ASTM Headquarters.

TABLE I

Chemical Analyses of the Test Cement

	By Calaveras Cement Co.	By California Div. of Highways
SiO ₂	23.92	23.8
Al ₂ O ₃	5.02	4.6
Fe ₂ O ₃	2.56	2.8
CaO	64.80	64.6
MgO	1.48	1.56
SO ₃	1.00	1.11
Ig. Loss	0.76	1.22
Insol. Res.	0.16	0.10
Na ₂ O	0.37	0.40
K ₂ O	0.29	0.26
Na ₂ O Equiv.	0.56	0.57
C ₄ AF	8	8
C ₃ A	9	8
C ₃ S	42	44
C ₂ S	37	35

TABLE II (a)

Mix 1:0.5

Batch Number	24-hour Compressive Strength SO ₃ in Cement				
	1.0	1.5	2.0	2.5	3.0
1a	3350	4494	5389	4812	4601
1b	3085	4654	5385	4732	4563
2a	3275	4517	5567	4967	4400
2b	3158	4504	5229	4933	4671
3a	3551	4594	5443	5150	5015
3b	<u>3553</u>	<u>4638</u>	<u>5574</u>	<u>5499</u>	<u>5165</u>
Average	3329	4567	5431	5016	4736
Relative	0	1238	2102	1687	1407

Change in Strength due to Addition
of 0.5% SO₃

Batch Number	SO ₃ in Cement			
	1.0	1.5	2.0	2.5
1a	1144	895	-577	-211
1b	1569	731	-753	-169
2a	1242	1050	-600	-567
2b	1346	725	-296	-262
3a	1043	849	-293	-135
3b	<u>1085</u>	<u>936</u>	<u>- 75</u>	<u>-334</u>
Average	1238	864	-432	-280

Table II (b)

Mix 1:1

24-hour Compressive Strength

Batch Number	SO ₃ in Cement				
	1.0	1.5	2.0	2.5	3.0
1a	2640	3696	4181	3958	3552
1b	2610	3665	3992	3852	3315
2a	2271	3438	4092	3858	3496
2b	2392	3558	4029	3933	3583
3a	2728	3711	4400	4293	3971
3b	2710	3766	4508	4442	4023
Average	2558	3639	4200	4056	3657
Relative	0	1081	1642	1498	1099

Change in Strength due to Addition
of 0.5% SO₃

Batch Number	SO ₃ in Cement			
	1.0	1.5	2.0	2.5
1a	1056	485	-223	-406
1b	1055	327	-140	-537
2a	1167	654	-234	-362
2b	1166	471	- 96	-350
3a	983	689	-107	-322
3b	1056	742	- 66	-419
Average	1080	561	-144	-399

Table II (c)

Mix 1:2

Batch Number	24-hour Compressive Strength SO ₃ in Cement				
	1.0	1.5	2.0	2.5	3.0
1a	1139	1699	1922	1827	1627
1b	1165	1811	2131	1851	1688
2a	1100	1779	2096	1896	1683
2b	1116	1778	2071	1852	1681
3a	1355	1722	2128	1948	1817
3b	1245	1845	2100	1999	1808
Average	1187	1772	2075	1896	1717
Relative	0	586	888	709	531

Change in Strength due to Addition
of 0.5% SO₃

Batch Number	SO ₃ in Cement			
	1.0	1.5	2.0	2.5
1a	560	223	- 95	-200
1b	646	320	-280	-163
2a	679	317	-200	-213
2b	662	293	-219	-171
3a	367	406	-180	-131
3b	600	255	-101	-191
Average	586	302	-179	-178

Table III

Mix 1:2

Expansion in Water - 72 hr. minus 24 hr. SO ₃ in Cement				
1.0	1.5	2.0	2.5	3.0
0.0040	0.0032	0.0028	0.0077	0.0140
Contraction in Air 3 days minus 7 days SO ₃ in Cement				
1.0	1.5	2.0	2.5	3.0
0.0615	0.0448	0.0372	0.0413	0.0558
Change in Contraction due to Addition of 0.5% SO ₃ SO ₃ in Cement				
1.0	1.5	2.0	2.5	
-0.0167	-0.0076	0.0041	0.0145	

Table IV

Constants for Correlation of Increments of SO_3 (X)
and Compressive Strength (Y) of Cement Mortars

$$\text{General Equation} = Y = a + bX$$

Labo- ratory	Mix	Y Intercept (a)	Slope (b)	Coefficient of Correlation (r)	Standard Error of Estimate $S_{y.x}$
1	1:0.5	1247	-1224	0.8474	429
2	"	1299	-1292	0.9183	311
3	"	1130	- 994	0.9231	232
1,2,3	"	1226	-1170	0.8862	342
1	1:1	978	-1034	0.9831	107
2	"	1096	-1059	0.9734	139
3	"	1066	- 994	0.9781	118
1,2,3	"	1047	-1029	0.9742	134
1	1:2	548	- 562	0.9324	122
2	"	609	- 620	0.9423	123
3	"	489	- 481	0.9161	118
1,2,3	"	549	- 555	0.9275	124

Table V (a)

1:0.5 Mix
Optimum SO₃ Computed from Individual Pairs of Strength Tests

$$g = \frac{n}{-1170} - 0.25 \text{ "true" optimum SO}_3 = 2.30\%$$

Batch No.	SO ₃	n	n/-1170	g	Indicated Opt. SO ₃	Difference from 2.30
1a	1.0	+1144	-0.98	-1.23	2.23	0.07
1b	1.0	+1569	-1.34	-1.59	2.59	0.29*
2a	1.0	+1242	-1.06	-1.31	2.31	0.01
2b	1.0	+1346	-1.15	-1.40	2.40	0.10
3a	1.0	+1043	-0.89	-1.14	2.14	0.16
3b	1.0	<u>+1085</u>	-0.93	-1.18	<u>2.18</u>	<u>0.12</u>
Avg.		+1238			2.31	0.01
Std. Dev.		196			0.16	
1a	1.5	+ 895	-0.76	-1.01	2.51	0.21
1b	1.5	+ 731	-0.62	-0.87	2.37	0.07
2a	1.5	+1050	-0.90	-1.15	2.65	0.35*
2b	1.5	+ 725	-0.62	-0.87	2.37	0.07
3a	1.5	+ 849	-0.73	-0.98	2.48	0.18
3b	1.5	<u>+ 936</u>	-0.80	-1.05	<u>2.55</u>	<u>0.25</u>
Avg.		+ 864			2.49	0.19
Std. Dev.		125			0.11	
1a	2.0	- 577	+0.49	+0.24	1.76	0.46*
1b	2.0	- 753	+0.64	+0.39	1.61	0.31*
2 1a	2.0	- 600	+0.51	+0.26	1.74	0.54*
2 1b	2.0	- 296	+0.25	+0.00	2.00	0.30*
3 1a	2.0	- 293	+0.25	+0.00	2.00	0.30*
3 1b	2.0	<u>- 75</u>	+0.06	-0.19	<u>2.19</u>	<u>0.11</u>
Avg.		- 432			1.88	0.42
Std. Dev.		252			0.22	
1a	2.5	- 211	+0.18	-0.07	2.57	0.27*
1b	2.5	- 169	+0.14	-0.11	2.61	0.31*
2a	2.5	- 567	+0.48	+0.23	2.27	0.03
2b	2.5	- 262	+0.22	-0.03	2.53	0.23
3a	2.5	- 135	+0.12	-0.13	2.63	0.33*
3b	2.5	<u>- 334</u>	+0.29	+0.04	<u>2.46</u>	<u>0.16</u>
Avg.		- 280			2.50	0.20
Std. Dev.		157			0.13	

*difference exceeds 0.25

Table V (b)

Mix 1:1

Optimum SO_3 Computed from Individual Pairs of Strength Tests

$$g = \frac{n}{-1029} - 0.25 \text{ "true" optimum } SO_3 = 2.27\%$$

Batch No.	SO_3	n	$n/-1029$	g	Indicated Opt. SO_3	Difference from 2.30
1a	1.0	+1056	-1.03	-1.28	2.28	0.01
1b	1.0	+1055	-1.03	-1.28	2.28	0.01
2a	1.0	+1167	-1.13	-1.38	2.38	0.11
2b	1.0	+1166	-1.13	-1.38	2.38	0.11
3a	1.0	+ 983	-0.96	-1.21	2.21	0.06
3b	1.0	+1056	-1.03	-1.28	2.28	0.01
Avg.		+1080			2.30	0.03
Std. Dev.		72			0.07	
1a	1.5	+ 485	-0.44	-0.69	2.19	0.08
1b	1.5	+ 327	-0.32	-0.57	2.07	0.20
2a	1.5	+ 654	-0.64	-0.89	2.39	0.12
2b	1.5	+ 471	-0.46	-0.71	2.21	0.06
3a	1.5	+ 689	-0.67	-0.92	2.42	0.15
3b	1.5	+ 742	-0.72	-0.97	2.47	0.20
Avg.		+ 561			2.29	0.01
Std. Dev.		159			0.16	
1a	2.0	- 223	+0.22	-0.03	2.03	0.24
1b	2.0	- 140	+0.14	-0.11	2.11	0.16
2a	2.0	- 234	+0.23	-0.02	2.02	0.25
2b	2.0	- 96	+0.09	-0.16	2.16	0.11
3a	2.0	- 107	+0.10	-0.15	2.15	0.12
3b	2.0	- 66	+0.06	-0.19	2.19	0.08
Avg.		- 144			2.11	0.18
Std. Dev.		69			0.07	
1a	2.5	- 406	+0.39	+0.14	2.36	0.09
1b	2.5	- 537	+0.52	+0.27	2.23	0.04
2a	2.5	- 362	+0.35	+0.10	2.40	0.13
2b	2.5	- 350	+0.34	+0.09	2.41	0.14
3a	2.5	- 322	+0.31	+0.06	2.44	0.17
3b	2.5	- 419	+0.41	+0.16	2.34	0.07
Avg.		- 399			2.36	0.09
Std. Dev.		76			0.07	

Table V (c)

Mix 1:2

Optimum SO₃ Computed from Individual Pairs of Strength Tests

$$g = \frac{n}{-555} - 0.25 \text{ "true" optimum SO}_3 = 2.24\%$$

Batch No.	SO ₃	n	n/-555	g	Indicated Opt. SO ₃	Difference from 2.24
1a	1.0	+560	-1.01	-1.26	2.26	0.02
1b	1.0	+646	-1.16	-1.41	2.41	0.17
2a	1.0	+679	-1.22	-1.47	2.47	0.23
2b	1.0	+662	-1.19	-1.44	2.44	0.20
3a	1.0	+367	-0.66	-0.91	1.91	0.33*
3b	1.0	+600	-1.08	-1.33	2.33	0.09
Avg.		+586			2.30	0.06
Std. Dev.		116			0.21	
1a	1.5	+223	-0.40	-0.65	2.15	0.09
1b	1.5	+320	-0.58	-0.83	2.33	0.09
2a	1.5	+317	-0.57	-0.82	2.32	0.08
2b	1.5	+293	-0.53	-0.78	2.28	0.04
3a	1.5	+406	-0.73	-0.98	2.48	0.20
3b	1.5	+255	-0.46	-0.71	2.21	0.03
Avg.		+302			2.29	0.05
Std. Dev.		63			0.25	
1a	2.0	-95	+0.17	-0.08	2.08	0.16
1b	2.0	-280	+0.50	+0.25	1.75	0.49*
2a	2.0	-200	+0.36	+0.11	1.89	0.35*
2b	2.0	-219	+0.39	+0.14	1.86	0.38*
3a	2.0	-180	+0.32	+0.07	1.93	0.31*
3b	2.0	-101	+0.18	-0.07	2.07	0.17
Avg.		-179			1.93	0.31
Std. Dev.		71			0.13	
1a	2.5	-200	+0.36	+0.11	2.39	0.15
1b	2.5	-163	+0.29	+0.04	2.46	0.22
2a	2.5	-213	+0.38	+0.13	2.37	0.13
2b	2.5	-171	+0.31	+0.06	2.44	0.20
3a	2.5	-131	+0.24	-0.01	2.52	0.28*
3b	2.5	-191	+0.34	+0.09	2.41	0.17
Avg.		-178			2.43	0.19
Std. Dev.		30			0.05	

* difference exceeds 0.25

Table VI

Optimum SO₃ Computed from Individual Pairs
of contraction tests

$$g = \frac{r}{0.021} - 0.25 \quad \text{True optimum SO}_3 = 2.07\%$$

Lab	SO ₃	Change in Contraction r	r/.021	g	Indicated Opt. SO ₃	Difference from 2.07
1	1.0	-.0167	-0.79	-1.04	2.04	0.03
	1.5	-.0076	-0.36	-0.61	2.11	0.04
	2.0	+.0041	+0.19	-0.06	2.06	0.01
	2.5	+.0145	+0.69	+0.44	2.06	0.01

Table VII

Precision of Compressive Strength Tests

Values are in units of psi computed from change in strength produced by increasing the SO₃ content 0.5 per cent above the increments shown in each column.

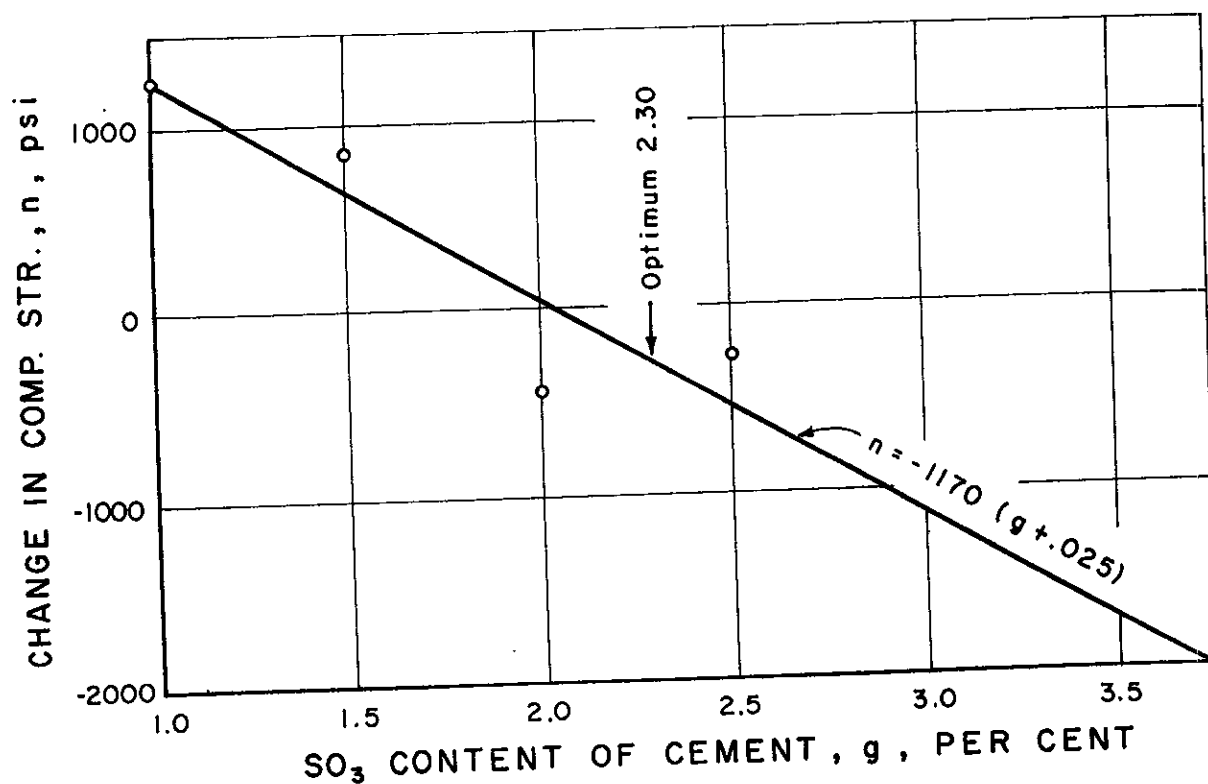
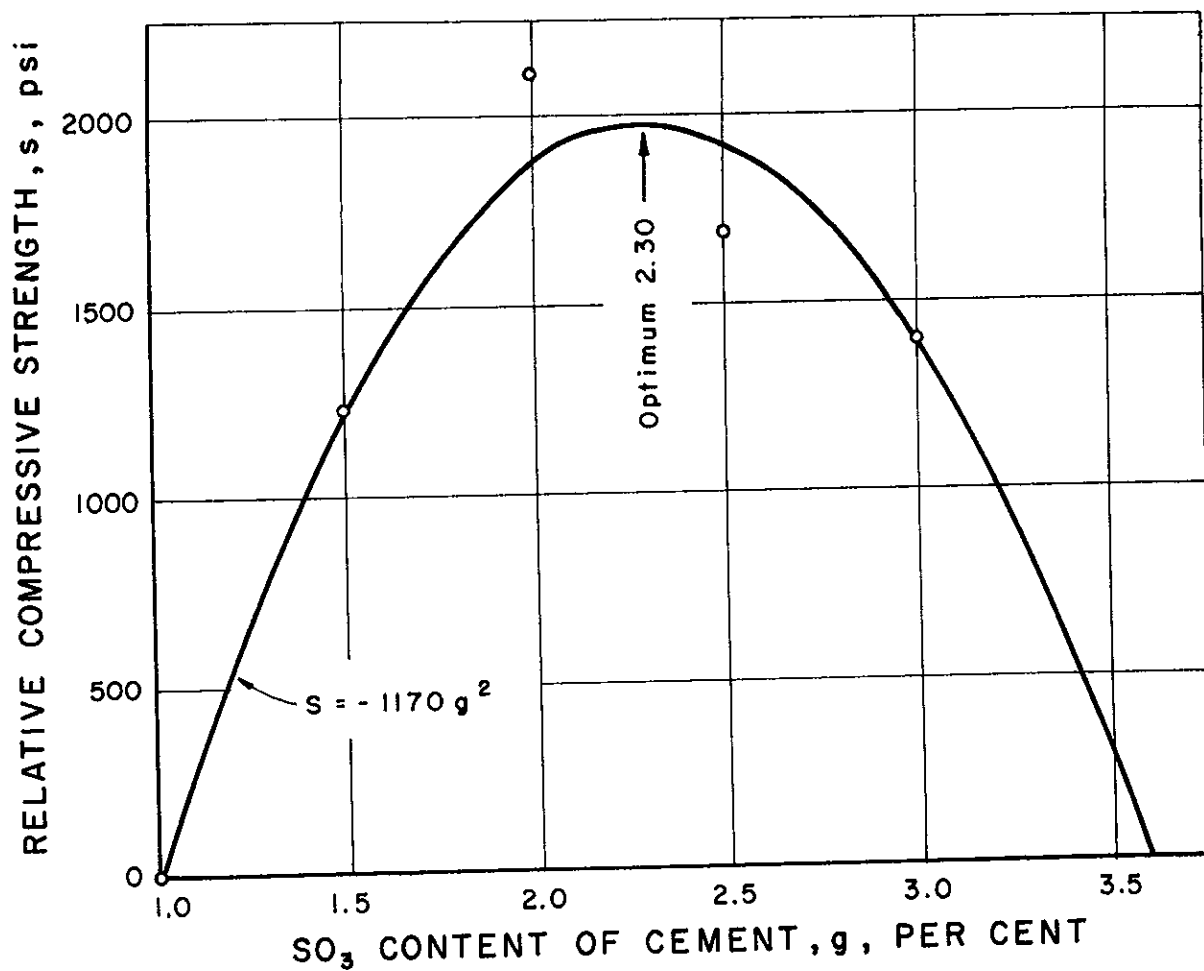
Mix 1:0.5						
SO ₃ in Cement	1.0	1.5	2.0	2.5	Avg.	Precision Ratio
Repeatability	621	529	719	518	597	1.02
Reproducibility	936	271	1465	723	849	1.46
Mix 1:1						
SO ₃ in Cement	1.0	1.5	2.0	2.5	Avg.	Precision Ratio
Repeatability	100	349	235	231	229	0.44
Reproducibility	466	941	309	309	506	0.98
Mix 1:2						
SO ₃ in Cement	1.0	1.5	2.0	2.5	Avg.	Precision Ratio
Repeatability	352	158	285	116	228	0.84
Reproducibility	575	179	215	96	266	0.96

Precision of Contraction Tests,
abstracted from reference (2)

		Precision Ratio
Repeatability	0.0055	0.81
Reproducibility	0.0046	0.67

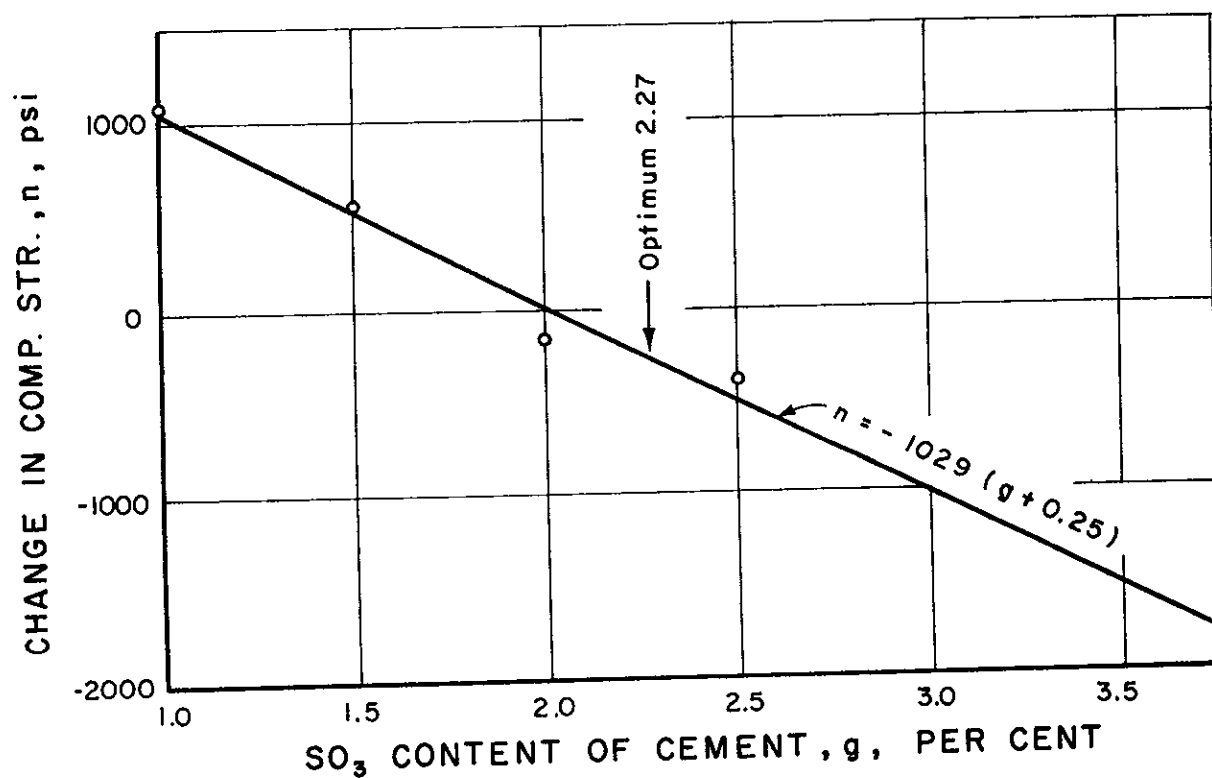
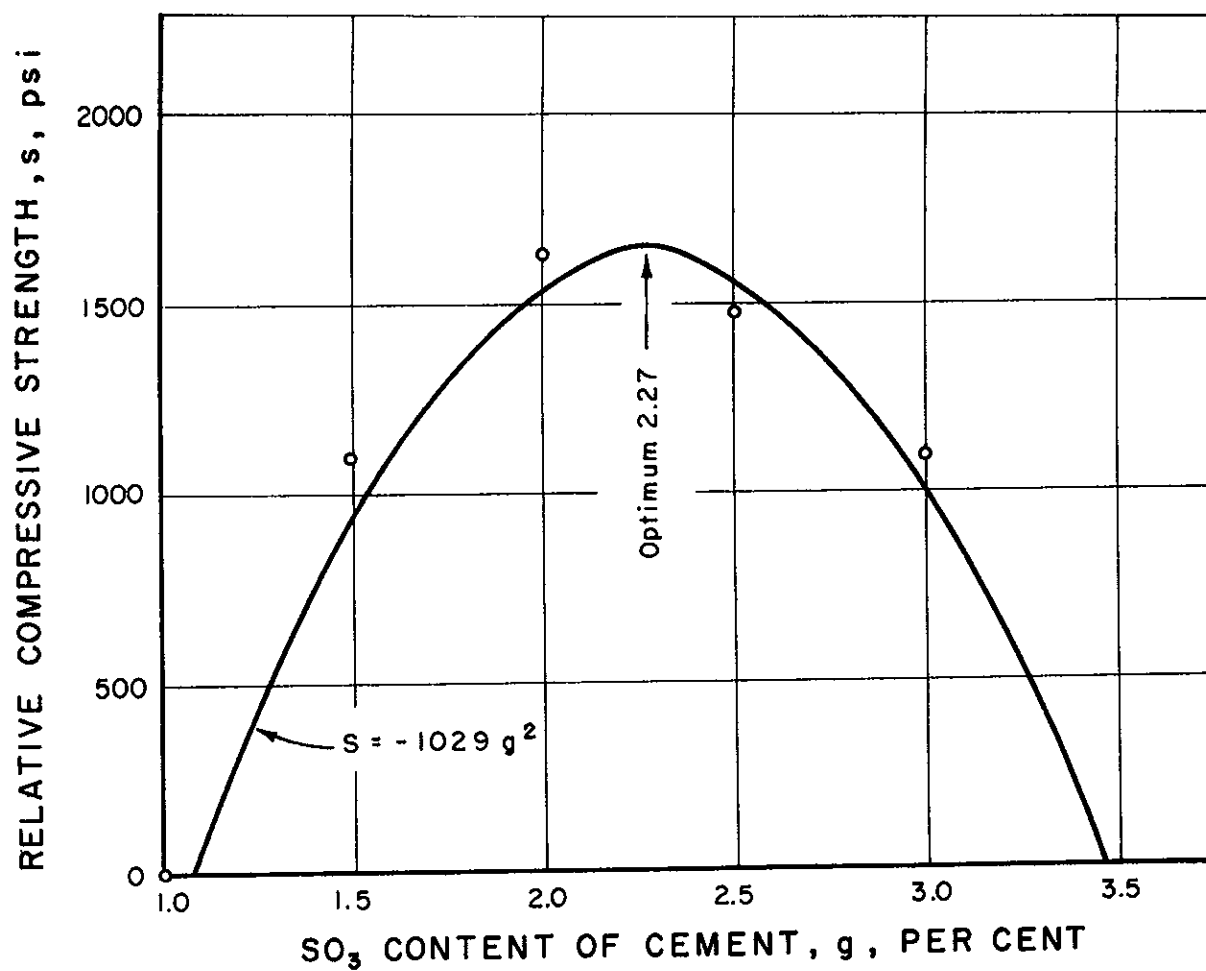
Note: Precision Ratio is the value of repeatability or reproducibility divided by one-half the coefficient of the applicable rate-of-change equation.

Fig. 1



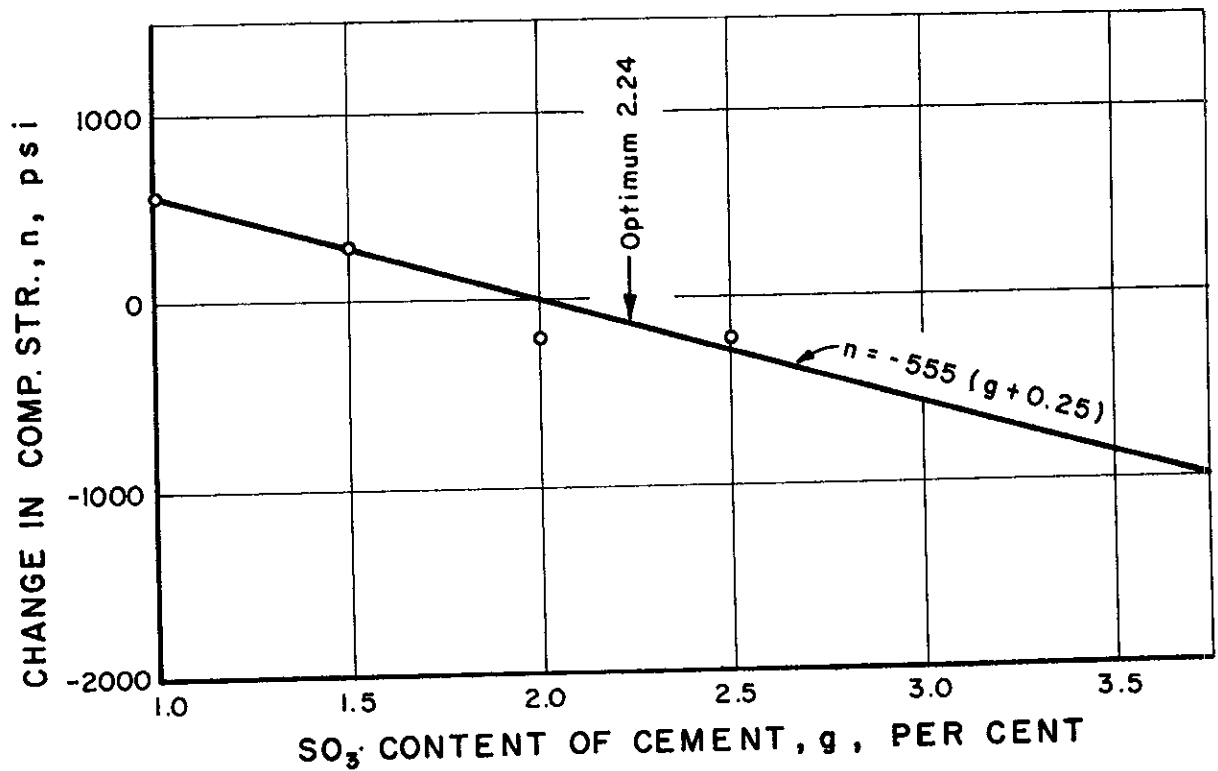
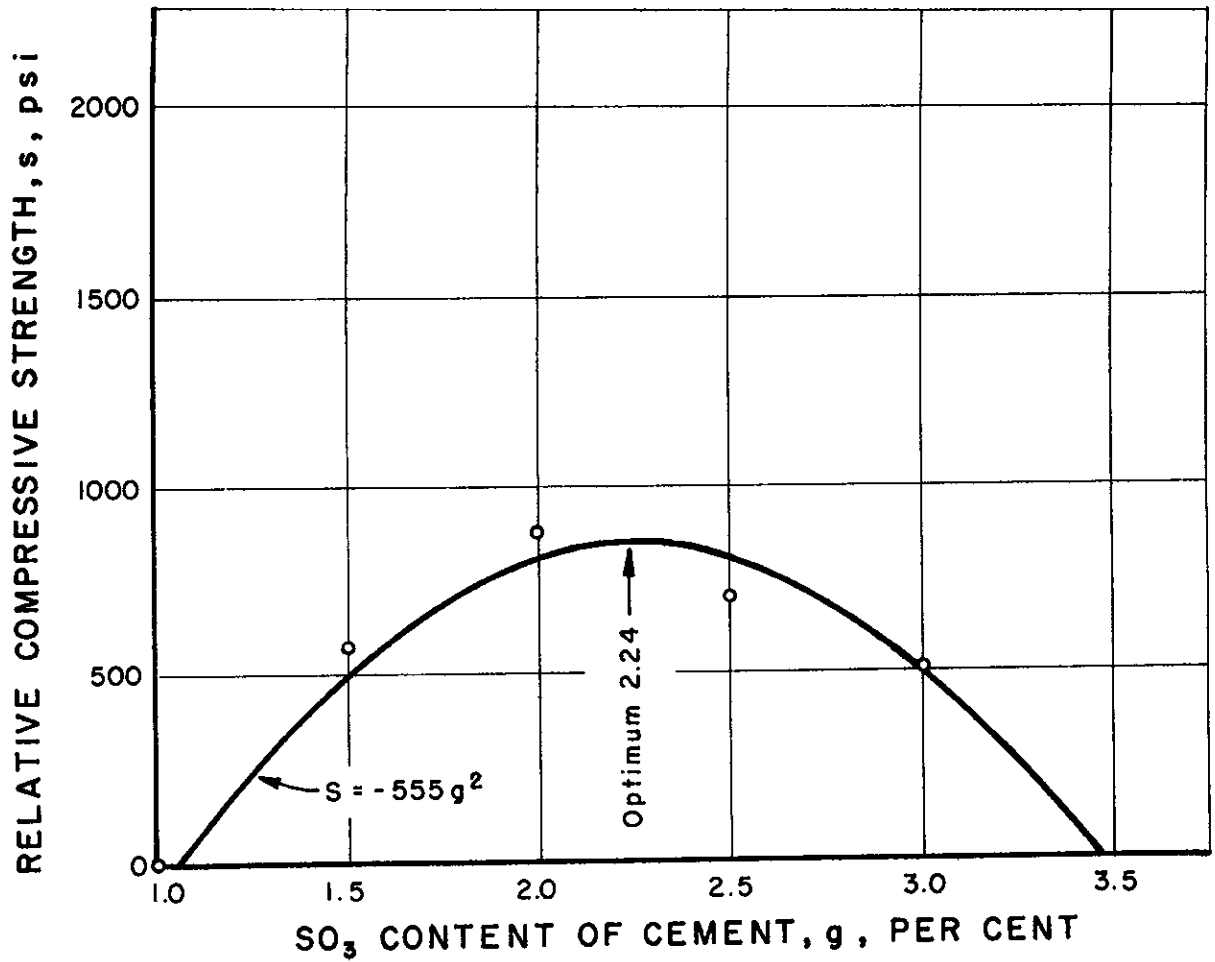
STRENGTH CURVES - MIX 1.0:0.5

Fig. 2



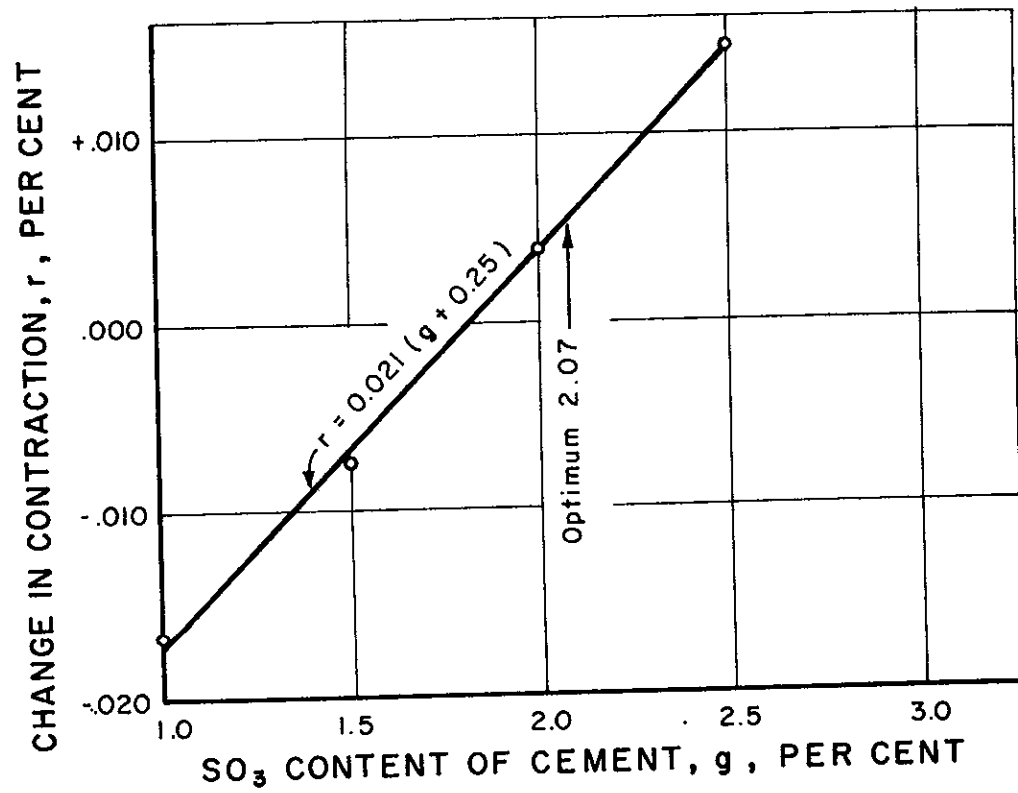
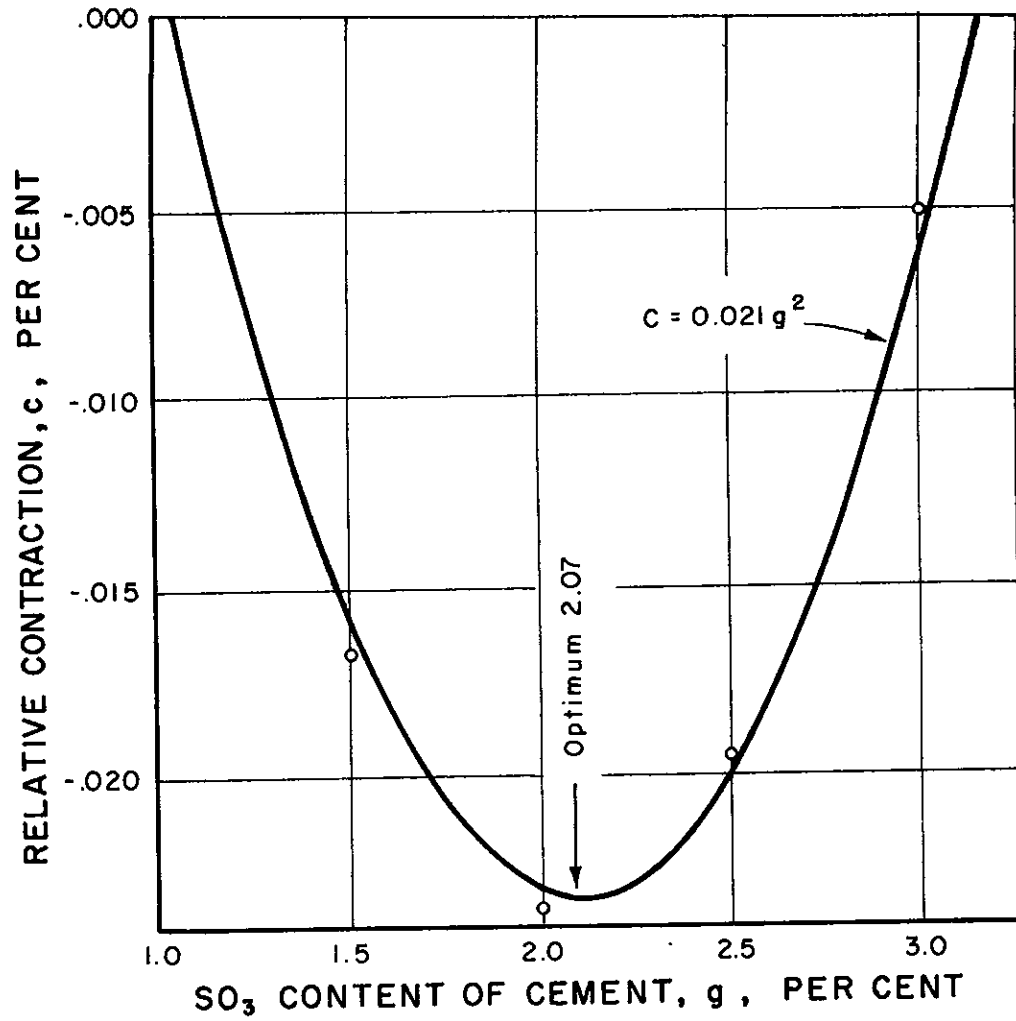
STRENGTH CURVES - MIX 1.0:1.0

Fig. 3



STRENGTH CURVES - MIX 1.0 : 2.0

Fig. 4



CONTRACTION TESTS - MIX 1.0: 2.0

APPENDIX

Tests of 2-in. Mortar Cubes at 24-hr Age to Indicate Optimum SO₃

Purpose

1. The purpose of this program is to determine if 2-in. mortar cubes tested at 24 hr. for compressive strength will reflect small changes in SO₃ content with sufficient reliability and precision to be used for indication of optimum SO₃ content.

Scope

2. Three cements (type I, II, and III) fairly different in C₃A, alkali content, and fineness are to be used. Each participant will work with a single cement. Two rounds of cubes will be made by each laboratory. One round will consist of five sets of six cubes each, with SO₃ contents varied by addition of gypsum in order to produce increments of SO₃ ranging from approximately 1 per cent below to 1 per cent above by 0.5 per cent steps. In order to bracket the indicated optimum more closely, two rounds of three supplemental batches each will be made consisting of indicated optimum, 0.25 per cent below and 0.25 per cent above.

Materials

3. a. The cements will be furnished by those shown and will be of the general composition indicated. All will be deficient in SO₃ by about 1 per cent as manufactured. The suppliers of the cements will also furnish complete analysis. The cements follow:

1. High-alkali high-C₃A type I, to be furnished by Dr. Hansen (A).
2. High-C₃A low-alkali type III, to be furnished by Mr. McCoy (B).
3. Low-C₃A low-alkali type II, to be furnished by Mr. Tremper (C).

b. Other materials are:

1. Gypsum, "Terra Alba," to be furnished by Mr. Offutt.
2. Standard Ottawa Sand
3. Graded Ottawa Sand

c. Two bags of cement and a gallon can of gypsum will be furnished each participant.

Tests

4. a. Cements will be tested by the laboratories shown:

<u>Cement</u>	<u>Laboratory</u>
A	Universal Atlas
A	Waterways Experiment Station
B	Portland Cement Association
B	Lehigh
B	Medusa
C	California Div. of Highways
C	Marquette
C	Ideal

b. The mortars will be of the proportions shown in
d. with the specimens made according to the applicable portions of C 109, and mixed by machine according to C 305.

c. All five or three batches for a round will be made on the same day and in as nearly consecutively as possible. Cube molds should be in uniformly good condition as substandard molds will result in poor strength that could be falsely attributed to improper SO_3 content. All the Ottawa sand for all batches of a single round should be blended prior to batching so that it will be uniform in grading and tendency to entrain air. All cement should be stored in airtight metal containers and it would be desirable to thoroughly blend both bags together before putting into the storage containers. Temperature of the room in which the specimens are made should remain uniform within ± 1 F during the entire time required for making the round. Temperature of the water and other materials when mixed should be identical for all batches in a round. All specimens for a round should be stored adjacent to each other in the curing room or cabinet and all should experience the same temperature history. Each set of specimens should be removed from the molds at 24 - 0.25 hr, placed in a pan of water at room temperature, wiped with a cloth and broken at 24 hr \pm 5 min. from the time water and cement first made contact.

- d. Mortar proportions and weights follow:

<u>Type I or II Cement</u>		
<u>1:1 Mortar</u>	<u>Material wt. g.</u>	<u>1:0.5 Mortar</u>
940	Cement	1200
470	Graded Ottawa Sand	300
470	Standard Ottawa Sand	300
282 (30%)	Water	324 (27%)

Type III Cement

<u>1:1 Mortar</u>	<u>Material wt. g.</u>	<u>1:0.5 Mortar</u>
940	Cement	1200
470	Graded Ottawa Sand	300
470	Standard Ottawa Sand	300
301 (32%)	Water	360 (30%)

e. The amount of water shown is approximate and was the amount giving flow between 110-115 per cent on experimental batches in the WES laboratory. The laboratory listed first for testing each cement will determine the amount of water to use for its cement and will inform the other labs using the same cement. Each lab will then use the determined amounts of water without change, but will determine and record flow.

f. Gypsum should be weighed separately from the other materials to the nearest 0.01 g. The gypsum should be considered as cement and the weight of cement decreased by the amount of added gypsum. The gypsum should be put in the water in the mixing bowl immediately before the addition of the cement.

g. The amount of gypsum to add will depend on the SO₃ content of the gypsum. Mr. Offutt will provide information on the analysis of the gypsum so that the participants in the program will not be required to make analyses.

h. The amount of gypsum to add will be computed by the method shown in Mr. Tremper's report, "Short-Time Tests of Mortars for Controlling SO₃ in Portland Cement at Optimum Value," Appendix p. 2.

$$X = \frac{A}{C-B} \times D$$

$$X = \text{g. gypsum in blend}$$

A = increase in SO₃ per cent to be obtained in blend

B = basic SO₃ content of cement

C = SO₃ content of gypsum per cent

D = wt g. of blended cement plus gypsum

An example follows:

Assume the cement as received contains 1 per cent SO_3 , the gypsum contains 40 per cent SO_3 , and it is desired to make a blend of cement and gypsum containing 1.5 per cent SO_3 , and the total amount of blend for a batch equals 1200 g.

$$\begin{array}{rcl} A & = & 0.5 \\ B & = & 1.0 \\ C & = & 40.0 \\ D & = & 1200.0 \end{array} \quad \begin{array}{l} \mathcal{X} = \frac{0.5}{40.0-1.0} \times 1200 \\ \mathcal{X} = 15.38 \text{ g. gypsum to add} \end{array}$$

$1200 - 15.38 = 1184.62$ g. cement before adding gypsum to raise SO_3 by 0.5 per cent.

Report

5. Each laboratory will record and tabulate its data sending one set promptly to the chairman with appropriate information on cement, temperature of making and curing, flow, time between contact of water and cement and test, indicated optimum SO_3 and any other relevant data.